MASTER COPY KEEP THIS COPY FOR REPRODUCTION PURPOSES

AD-A271 290

ON PAGE

Form Approved
OMB No. 0704-0188

Public ... gather... collecti... Davis ... hour per response, including the time for reviewing instructions, searching existing data sources, ... collection of information. Send comments regarding this burden estimate or any other aspect of this ... hington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson ... agement and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

1. A	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	9-28-93	Final 5/1/92 - 8/31/93

4. TITLE AND SUBTITLE

Nanofabrication by Focused Ion Beams

5. FUNDING NUMBERS

(2)

DAAL03-92-G-0217

6. AUTHOR(S)

Dr. John Melngailis
Prof. Dimitri A. Antoniadis

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

Research Laboratory of Electronics
Massachusetts Institute of Technology
77 Massachusetts Avenue
Cambridge, MA 02139

8. PERFORMING ORGANIZATION REPORT NUMBER

DTIC ELECTE OCT 21 1993 S A D

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

U. S. Army Research Office
P. O. Box 12211
Research Triangle Park, NC 27709-2211

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

ARO 29631.2-EL

11. SUPPLEMENTARY NOTES

The view, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy, or decision, unless so designated by other documentation.

12a. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release; distribution unlimited.

12b. DISTRIBUTION CODE

93-24944

13. ABSTRACT *(Maximum 200 words)*

Nanometer scale patterning is one of the keys to advances in electronic devices. At present most patterning is done by a multistep lithography process: resist is exposed and developed and the uncovered surface is altered. The focused ion beam may offer simplification of this multistep process. A surface film may be made to grow or dissolve where it has been irradiated. This can be viewed as a new (inorganic) resist process, but in some cases the patterned film may be the desired final structure on the substrate. The problem is that,

(continued on reverse side)

14. SUBJECT TERMS

Nanofabrication, Focused Ion Beams, Ion Sensitive Films, Semiconductors, Quantum Effect Devices

15. NUMBER OF PAGES

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	UL

although examples of such ion sensitive films have been found, they suffer from some drawback such as low sensitivity, or poor resolution.
One of the aims of the work was to seek new and useful processes and materials.

Another direct pattering technique, more ideally suited for ion beams, is direct maskless implantation of dopants in semiconductors. One of the limits of resolution of this technique is the lateral straggle of ions as they penetrate into the substrate. This has important bearing on the ability to fabricate the confined carrier structures in semiconductors needed in the study of quantum effect devices. The other object of the work was to characterize this straggle and to apply focused ion beam implantation to the fabication of confined carrier structures, in particular the in-plane-gate field effect transistor. The final report provides a summary of the most important results.

"Nanofabrication by Focused Ion Beams"

FINAL REPORT

John Melngailis and Dimitri A. Antoniadis

September 22, 1993

to

U.S. Army Research Office

Grant Number DAAL 03-92-G-0217

Massachusetts Institute of Technology
Cambridge, MA 02139

APPROVED FOR PUBLIC RELEASE;

DISTRIBUTION UNLIMITED

I. STATEMENT OF THE PROBLEM

Nanometer scale patterning is one of the keys to advances in electronic devices. At present most patterning is done by a multistep lithography process: resist is exposed and developed and the uncovered surface is altered. The focused ion beam may offer simplification of this multistep process. A surface film may be made to grow or dissolve where it has been irradiated. This can be viewed as a new (inorganic) resist process, but in some cases the patterned film may be the desired final structure on the substrate. The problem is that, although examples of such ion sensitive films have been found, they suffer from some drawback such as low sensitivity, or poor resolution. One of the aims of our work was to seek new and useful processes and materials.

Another direct patterning technique, more ideally suited for ion beams, is direct maskless implantation of dopants in semiconductors. One of the limits of resolution of this technique is the lateral straggle of ions as they penetrate into the substrate. This has important bearing on the ability to fabricate the confined carrier structures in semiconductors needed in the study of quantum effect devices. The other object of our work was to characterize this straggle and to apply focused ion beam implantation to the fabrication of confined carrier structures, in particular the in-plane-gate field effect transistor..

II. SUMMARY OF MOST IMPORTANT RESULTS

A. Focused ion beam patterning

We have collaborated with researchers at Lincoln Laboratory at the Naval Research Laboratory and at MIT in exploring novel films for ion beam patterning. The films have ranged from inorganic, plasma-grown oxide to monolayer structures. The films were exposed using our focused ion beam test pattern consisting of 10 x 10 μm areas at an array of doses as well as single-pass, minimum linewidth features. The squares were used to measure the sensitivity

and the small features were used to determine the resolution. Generally, Au, Si, Be, and Ga ions beams were used at energies ranging from 60 to 280 keV.

1. Tungsten Oxide Film, WO_3. (In collaboration with A. Forte, and M. Rothschild of Lincoln Laboratory).

WO_3 films about 50 nm thick were grown over polyimide or AZ resist using plasma and a mixture of WF_6, H_2 and O_2 gases. The films were then exposed with the test pattern using Si, Be, and Ga ions. The focused ion beam rendered the film insoluble in a CF_4 plasma etch provided the dose was higher than 10^{15} ions/cm^2. After the CF_4 "development" the pattern was transferred to the underlying organic film with an O_2 plasma etch. The minimum dimensions obtained were 0.2 to 0.3 μm. The attractive feature of this "resist" film is the fact that it is all dry and therefore compatible with in-situ processing. The drawbacks are relative insensitivity (100x higher dose needed than for PMMA exposure), the modest resolution demonstrated and the fact that the plasma deposition of WO_3 is not yet well controlled. The process which makes the WO_3 sensitive to ions is at this point not understood. A more extensive study of the fundamental ion-oxide interaction appears to be needed to make progress in this area. This was beyond the scope of our research program.

2. Alteration of Monolayer Films

a) Electroless Metalization after Exposure to Ions. (In collaboration with J.M. Calvert, Naval Research Laboratory). A substrate surface is covered with a ligating monolayer film. Exposure to the ion beam disturbs the monolayer. Thus when electroless deposition of Pd, for example, is performed the Pd grows only on the unexposed areas. The minimum dose needed to produce patterns was 1 to 2.5×10^{14} ions/cm^2 when exposed with Si ions. The minimum linewidths observed so far are 0.2 to 0.3 μm, See Fig. 1. A manuscript for publication is being prepared by the NRL.

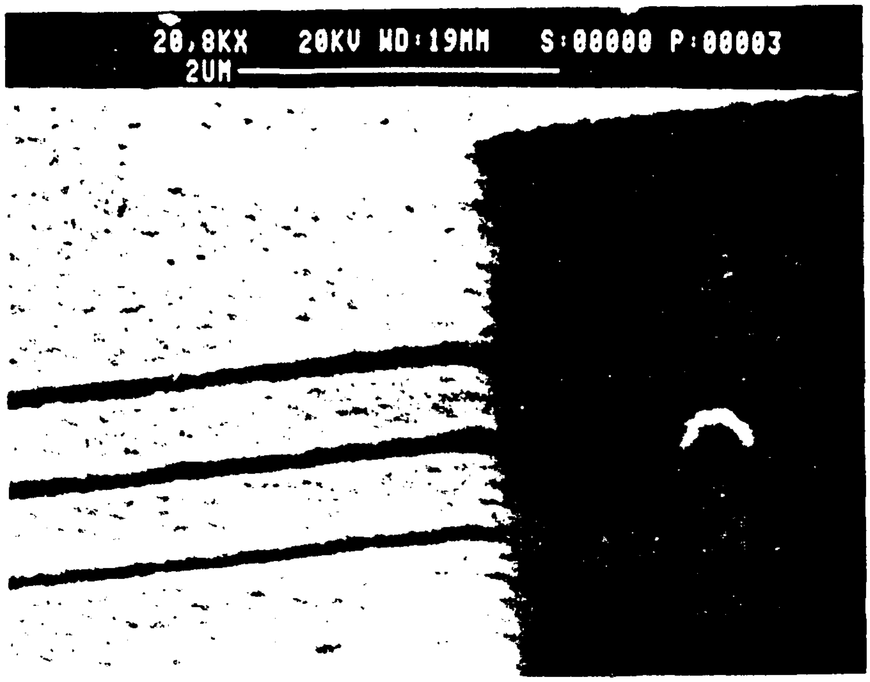


Fig. 1 Pd film deposited on the areas not exposed by the ion beam, i.e. the ion beam exposed a pad on the light with three lines going toward the left. The exposures were carried out with Si^{++} ions at 110 keV. The beam diameter at this (non optimum) voltage is of order 0.2 µm. Thus the line width observed is as expected. The sharp, well defined sidewalls indicate that much finer lithography would be possible with a more optimum beam.

b) Preferential Oxide growth after ion exposure. (In collaboration with R. Kunz Lincoln Laboratory). A silicon surface is covered with a monolayer terminating in CF_3. When this film is exposed to an ion beam, the termination changes to OH. Either SiO_2 or TiO_2 films can then be preferentially grown at room temperature on the exposed areas using alternate exposure to water vapor and metal chloride. Preferential oxide growth was observed for doses in excess of $5 \times 10^{13}/cm^2$ for Au ions, $10^{14}/cm^2$ for Si ions, and $5 \times 10^{15}/cm^2$ for Be ions. This process looks very promising particularly if it could be carried out in-situ, which is not possible with our apparatus.

c) Alkane thiol molecules. (In collaboration of Dan Frisbee of M. Wrighton's group Chemistry Dept. MIT). The alkane thiol molecule can be made to bond to gold (or other metals such as Cu, Pt, etc.) as a monolayer. Ion beam exposure removes this monolayer and the gold can then be preferentially etched in the exposed areas with a conventional wet etch. (This process is based on the work of G. Whiteside of Harvard). Gold was selected as the material to pattern because of a specific application for gold microelectrodes in chemistry. The sensitivity of this process was ~ 10^{16} ions/cm^2.

3. Telfon Films. (In collaboration with A. Forte and M. Rothschild of Lincoln Laboratory). In this process a commercial Teflon film (AF2600) is spun on the wafer. Ion exposure alters the properties of this film so that either a SiO_2 film can be grown selectively by a room temperature $H_2O + SiCl_4$ vapor process on the exposed area, or simply AZ resist can be spun on and will only stick on the exposed area. With Ca^+ ions at 80 keV the sensitivity of this process is 10^{14} - 10^{15} ions/cm^2. After the preferential coverage the unexposed teflon is etched away in an O_2 reactive ion etch and the pattern can be further transferred into the Si substrate. An example of a ~ 0.1 μm pattern is shown in Fig. 2.

FIB PATTERNING OF Si WITH TEFLON RESIST

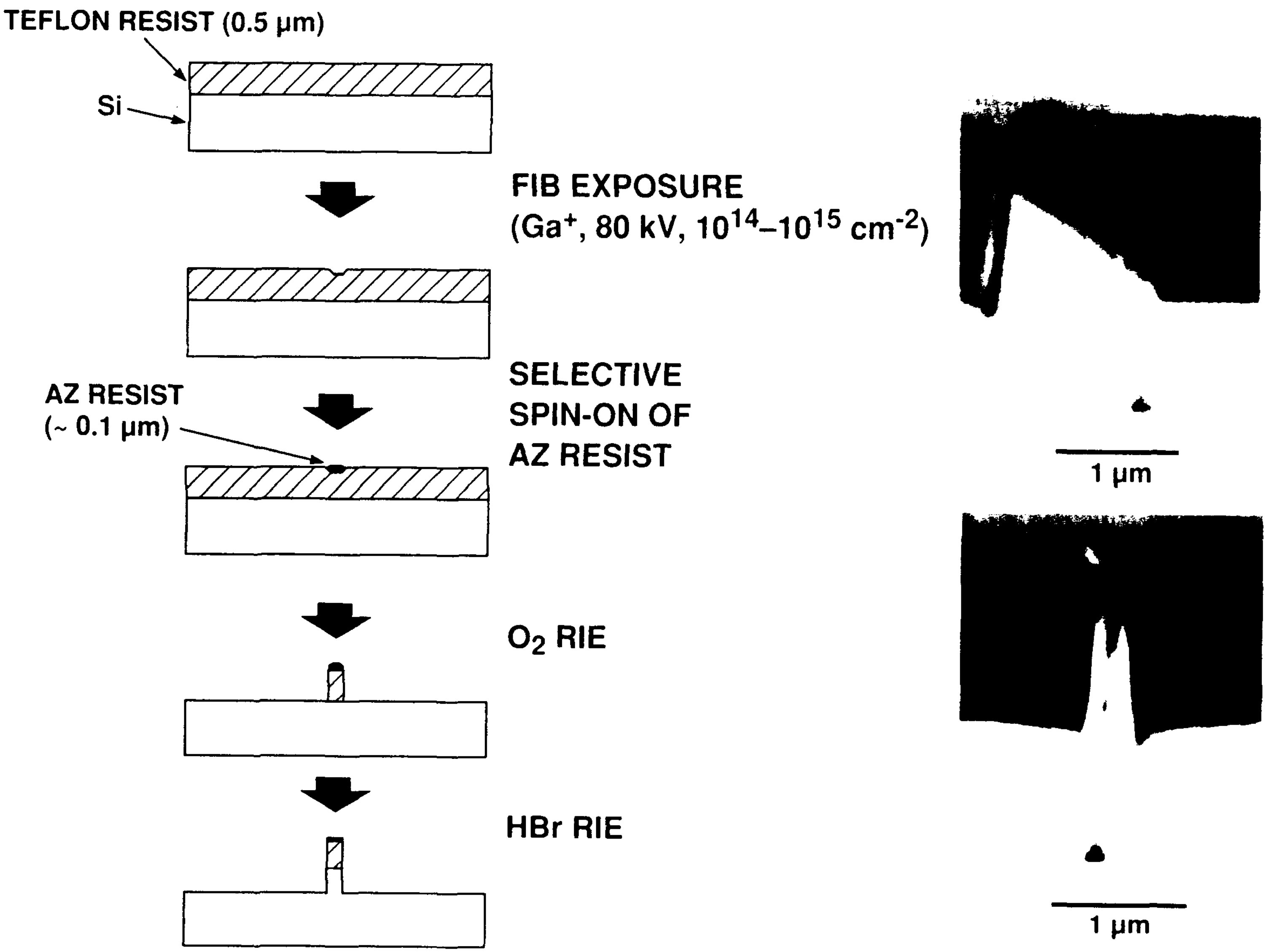
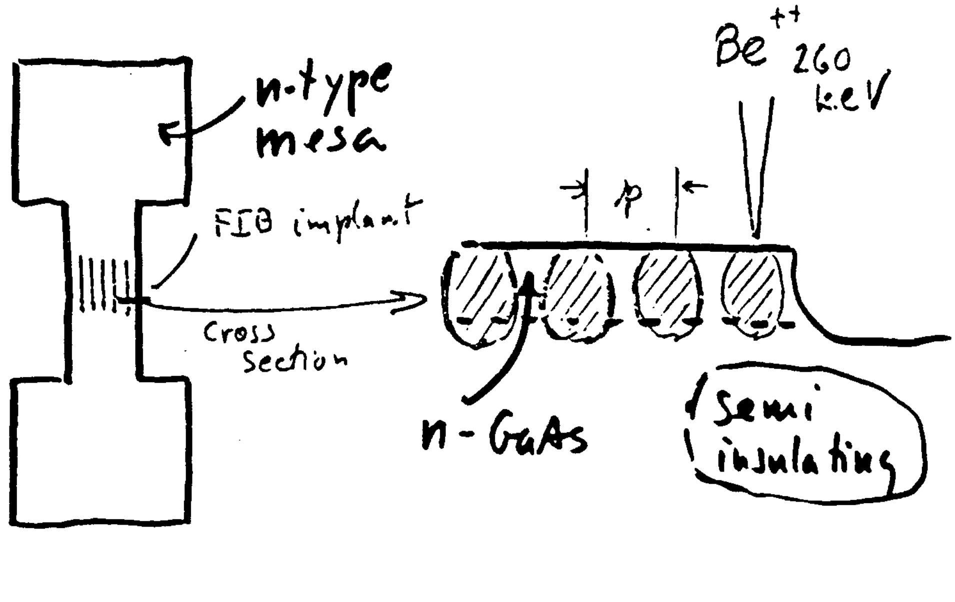
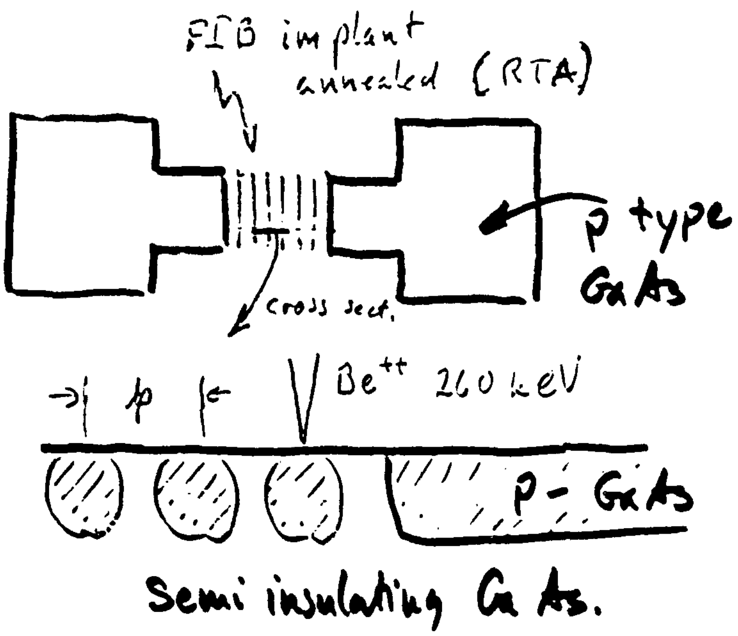


Fig. 2 A submicron Si line patterned using Teflon resist. The line width at the top is seen to be ~ 0.1 μm much finer lithography appears to be possible with a more optimum beam.

211952-1



3(a)



3(b)

Fig. 3(a) On the left, the top view of the n-type mesa on a semi-insulating GaAs substrate. Be stripes are implanted by the focused ion beam, as shown in cross section on the right. As the period of the grating lines, p, decreases the conduction is cut off by the implanted (damaged) regions, shown shaded.

3(b) Same as 3(a) but for the opposite polarity conduction. Here the sample is annealed (RTA) and conduction is <u>initiated</u> when the period p decreases and grating lines "touch".

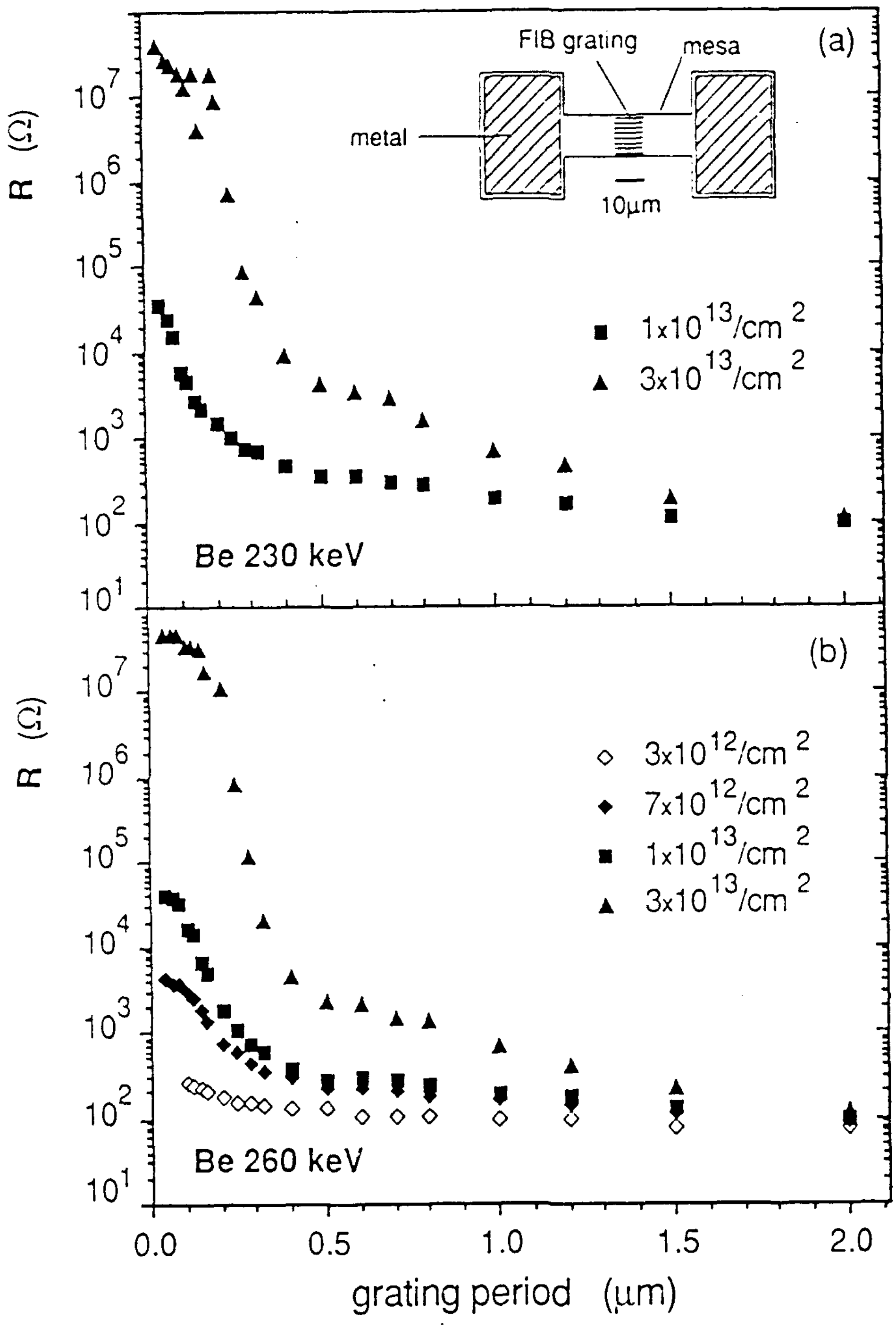


Fig. 4 The inset shows schematic of the mesa structure in an n-type GaAs epilayer as in Fig. 3(a). The implanted grating lines create insulating regions. Thus the resistance rises as the grating period is reduced. The plots show the resistance of the structure as a function of grating period for various doses at two energies.

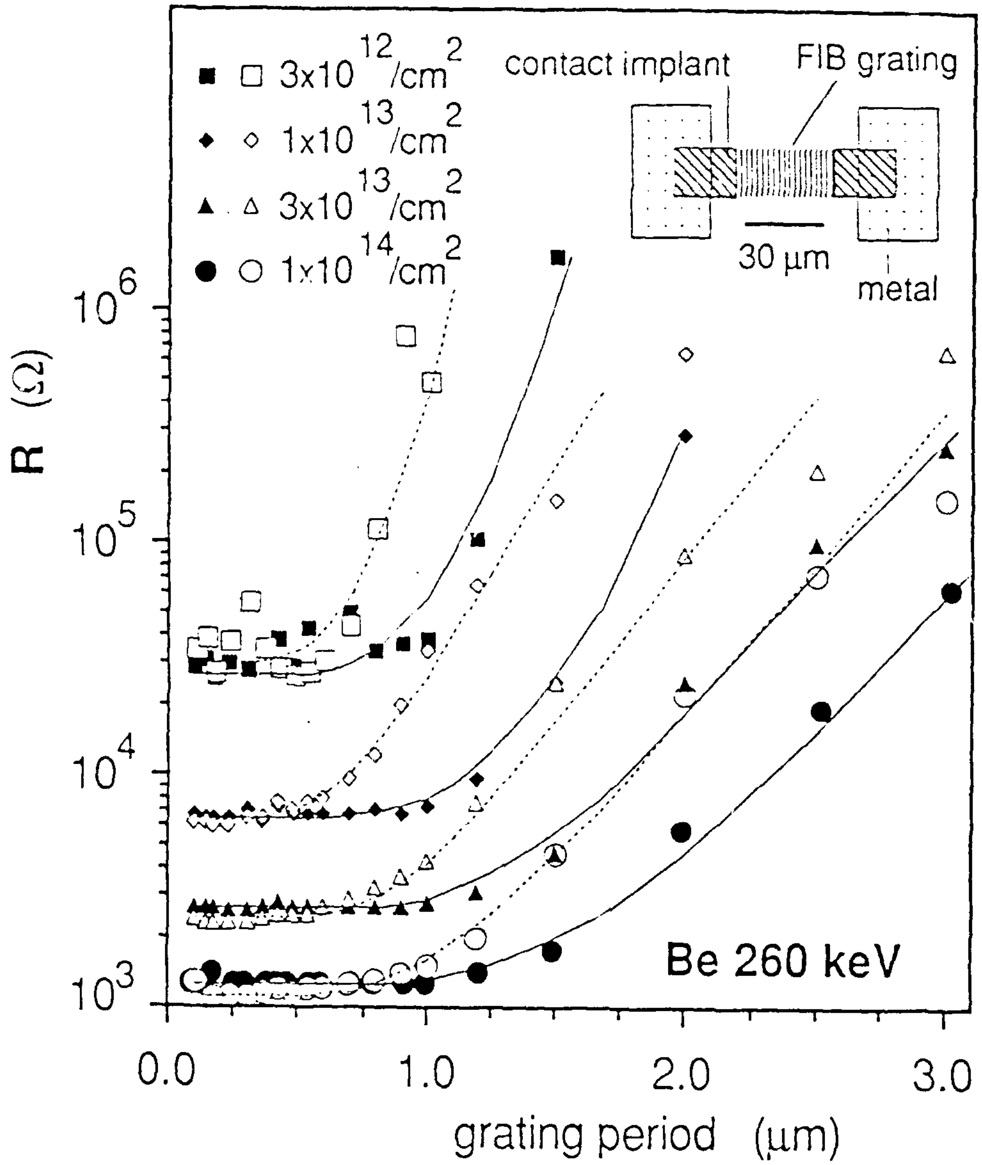


Fig. 5 The inset shows a schematic of the structure where two contacts are made to implanted pads in semi-insulating GaAs as in Fig. 3(b). A grating has been implanted between these pads over a 10 μm x 30 μm area with a focused Be^{++} ion beam at 260 keV. The resistance from pad to pad after annealing is plotted vs. the period of the grating. The filled symbols fitted by a model with a solid line are for off-axis implantation and the corresponding open ones fitted with dashed lines are for on-axis implantation at the same dose.

We have demonstrated the use of focused ion beams as a patterning tool for exposing oxide films, various self assembled monolayers, and teflon films. Some of these examples look quite promising. To make further progress would require a larger collaborative project between a focused ion beam group and a surface chemistry group.

B. Limited lateral straggle

In order to measure the lateral straggle of Be and Si ions implanted in GaAs we implanted gratings both parallel and perpendicular to the direction of current flow between two contacts. The period of the gratings was varied in order to determine when they "smear out". From this we deduced the lateral straggle. The geometry of the gratings is shown in Fig. 3. In the parallel geometry Be ions were implanted at 230 and 260 keV into n-type GaAs. With no anneal the implanted region becomes insulating. Thus when the period of the grating (Fig. 3(a)) decreases the conduction is eventually cut off. See Fig. 4. Since the dependence of resistivity on period has not become flat even below 0.1 μm, there is a modulation of the conduction even for those very small periods. (In these implantations the dose is normalized so that the total dose delivered in ions/cm^2 is constant, independent of the period.) From these experiments one can get a quantitative feel for the extent of the straggle.

We were able to deduce an actual number for the straggle in the perpendicular geometry (Fig. 3(b)). Here the grating is implanted with Be ions into an insulating region between two p type contacts. The devices are annealed so that conduction occurs when the implanted p-type grating lines begin to touch. The implantations were carried out both on and off axis and at 4 different average doses. The results are shown in Fig. 5. The off-axis implants clearly show an earlier onset of conduction indicating a larger lateral straggle. Using a simple model of a Gaussian distribution of carriers across each line of the grating,

we deduced the standard deviation. This standard deviation is the sum, in quadrature, of the initial (Gaussian) beam profile, the straggle, and the diffusion during anneal. We measured the initial beam profile by exposing lines in PMMA at various doses. The contribution of this to the standard deviation is 0.065 ± 0.015 μm thus the lateral straggle is reduced by this amount (in quadrature). The lateral straggle ΔR_t has been calculated to be 322 or 450 nm for 260 keV Be into GaAs. The measured value, however, is less than 190 nm for implantation off the symmetry axis and less than 140 nm on axis. Thus the lateral straggle is considerably smaller than expected. This will make applications of focused ion beams to the lateral confinement of carrier distributions much more promising.

C. <u>In-plane-gate field effect transistor</u>

(The work is largely the Masters Thesis of Mark Armstrong and is supported by AASERT funds under the parent grant).

The two dimensional electron gas in a GaAs/AlGaAs heterostructure can be divided up into islands by ion implantation. The ion bombardment renders conducting material insulating. The focused ion beam is a convenient tool for doing this, particularly since the lateral straggle is limited, as we have seen above, and since no mask is needed. The in-plane-gate field effect transistor (IPGFET) is of interest as a device and also as a vehicle for understanding the limits and uses of this technique. (Details and references to earlier literature are in M.A. Armstrong's MS Thesis). The configuration of the IPGFET is shown in Fig. 6. A number of these structures have been built with different geometric channel lengths L_{geo} and widths W_{geo}. The minimum electrical width of the insulating line is determined by the focused ion beam diameter, the lateral straggle and the depletion. The beam diameter and lateral straggle are as

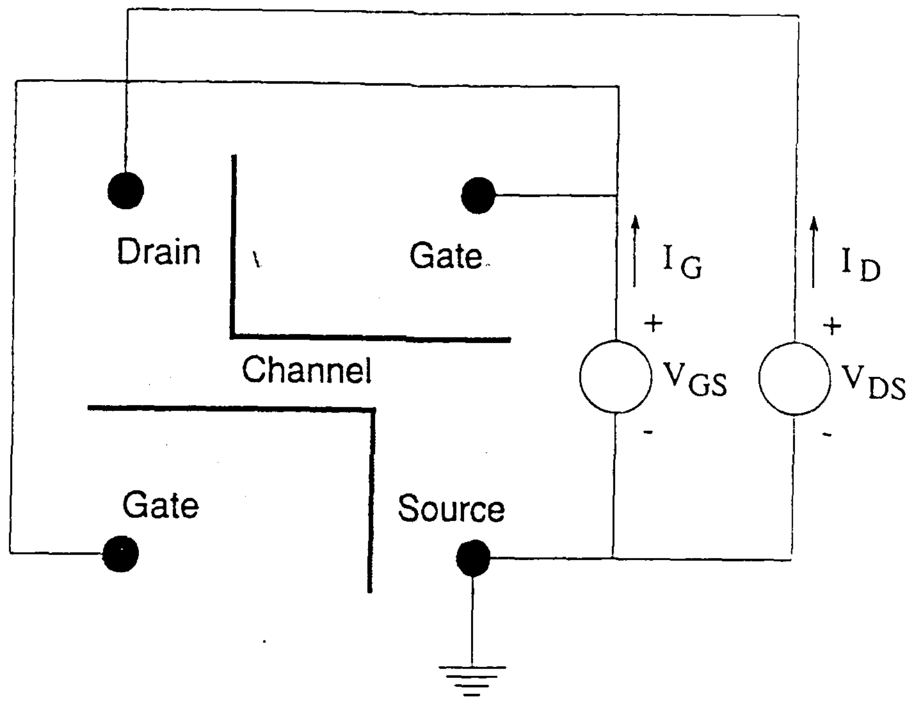


Fig. 6 Schematic of the in-plane-gate transistor. The focused ion beam implants are the two dark "L" shaped lines dividing the source-gate-drain regions.

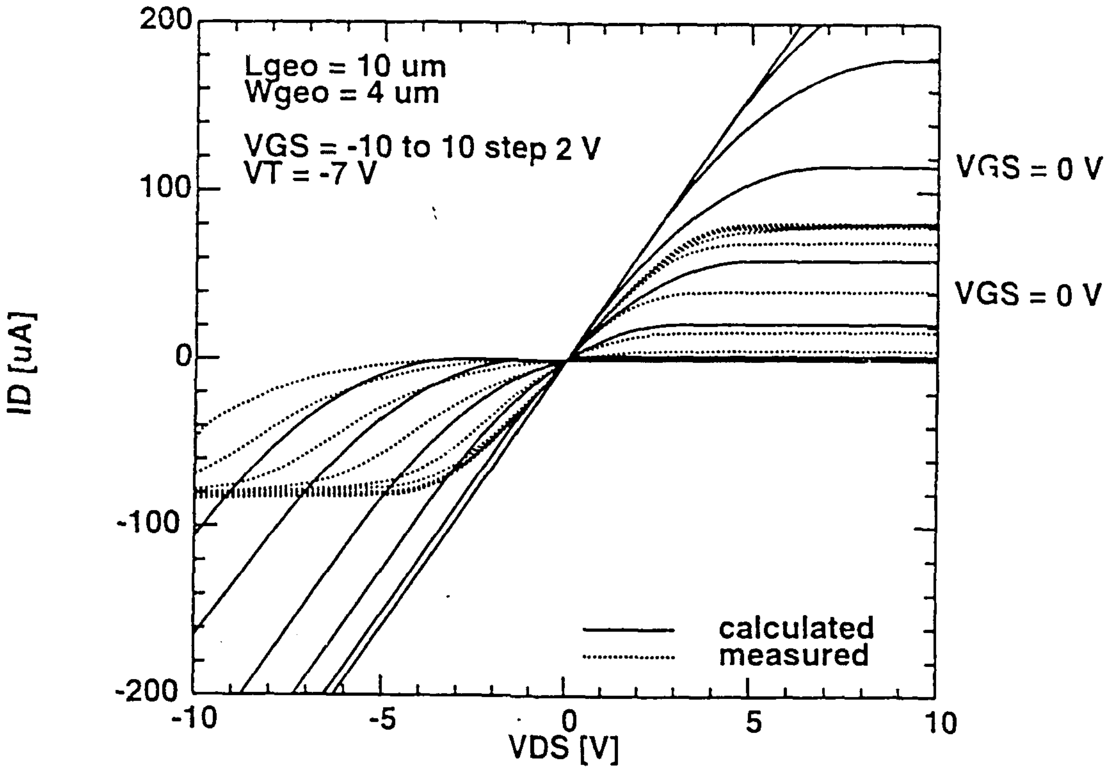


Fig. 7 Comparison of calculated and measured IV curves. The V_{GS} = 9 V line is labeled for both sets of curves.

reported above. By measuring the channel conductance as a function of geometric channel width W_{gco}. One can deduce the depletion width to be 0.75 μm, i.e. depletion adds 0.75 μm to each edge of the focused ion beam written line. the depletion or accumulation around the insulating line, and hence the capacitance, can be varied by applying a voltage from the source to the gate (V_{GS}) i.e. across the insulating line. A model of this capacitance as a function of V_{GS} has been developed and verified by experiment. In addition the transistor properties of the IPGFET have been measured and calculated. The results are shown in Fig. 7. The disagreement in the maximum I_D is due to velocity saturation which is not included in the model, i.e. in the ideal curves the current is unlimited, while the measured curves clearly saturate. Overall the IPGFET has been well characterized by our series of measurements. The model derived provides, for the first time, an understanding of its operation. The structures appear promising for use in quantum effect, one dimensional conductor, test structures.

III. LIST OF PUBLICATIONS

D. Vignaud, S. Etchin, K.S. Liao, C.R. Musil, D.A. Antoniadis, and J. Melngailis, "Lateral straggle of focused-ion-beam implanted Be in GaAs", Appl. Phys. Lett. 60, 2267 (1992).

D. Vignaud, C.R. Musil, S. Etchin, D.A. Antoniadis, and J. Melngailis, "Lateral straggle of Si and Be focused-ion-beam implanted in GaAs", J. Vac. Sci. Technol. B11, 581 (1993).

J. Melngailis, "Focused ion beam lithography", Nucl. Instr. and Methods in Physics Research B80/81, 1271 (1993) (Invited plenary paper at 8th International Conference on Ion Beam Modification of Materials, Heidelberg, Sept. 7-11, 1992).

A. Chu, H.M. Cronson, J.F. Devine, S. Soares, M.N. Solomon, H.J. Lezec, and C.R. Musil, "RF built-in test and enabling technologies for integrated diagnostics", IEEE Systems Readiness Conference and Automatic Testing Conference, Dayton, Ohio, Sept. 11-14, 1992.

M.A. Armstrong, MS Thesis MIT, Dept. of EECS (1993). "Characterization of In-Plane-Gate Field Effect Transistors".

IV. PERSONNEL WORKING ON THE PROJECT

- Mark Armstrong, Research Assistant, EE&CS,
  Supported by AASERT Grant no. DAAL 03-92-G-0305
  MS Degree – expected Sept. 1993

- Henri J. Lezec, Research Assistant, EE&CS,
  Ph.D. Degree – awarded June 1992

- Christian R. Musil, Research Assistant, EE&CS,
  Ph.D. Degree – awarded June 1993

- Sergey Etchin – Research Engineer

- Dominique Vignaud – Visiting Scientist
  (partial research support only)

- Akira Shimase – Visiting Scientist
  (partial research support only)

- Dimitri A. Antoniadis – Professor EE&CS

- John Melngailis – Senior Research Scientist

Fig. 1 Pd film deposited on the areas not exposed by the ion beam, i.e. the ion beam exposed a pad on the light with three lines going toward the left. The exposures were carried out with Si^{++} ions at 110 keV. The beam diameter at this (non optimum) voltage is of order 0.2 μm. Thus the line width observed is as expected. The sharp, well defined sidewalls indicate that much finer lithography would be possible with a more optimum beam.

Fig. 2 A submicron Si line patterned using Teflon resist. The line width at the top is seen to be ~ 0.1 μm much finer lithography appears to be possible with a more optimum beam.

Fig. 3(a) On the left, the top view of the n-type mesa on a semi-insulating GaAs substrate. Be stripes are implanted by the focused ion beam, as shown in cross section on the right. As the period of the grating lines, p, decreases the conduction is cut off by the implanted (damaged) regions, shown shaded.

3(b) Same as 3(a) but for the opposite polarity conduction. Here the sample is annealed (RTA) and conduction is <u>initiated</u> when the period p decreases and grating lines "touch".

Fig. 4 The inset shows schematic of the mesa structure in an n-type GaAs epilayer as in Fig. 3(a). The implanted grating lines create insulating regions. Thus the resistance rises as the grating period is reduced. The plots show the resistance of the structure as a function of grating period for various doses at two energies.

Fig. 5 The inset shows a schematic of the structure where two contacts are made to implanted pads in semi-insulating GaAs as in Fig. 3(b). A grating has been implanted between these pads over a 10 μm x 30 μm area with a focused Be^{++} ion beam at 260 keV. The resistance from pad to pad after annealing is plotted vs. the period of the grating. The filled symbols fitted by a model with a solid line are for off-axis implantation and the corresponding open ones fitted with dashed lines are for on-axis implantation at the same dose.

Fig. 6 Schematic of the in-plane-gate transistor. The focused ion beam implants are the two dark "L" shaped lines dividing the source-gate-drain regions.

Fig. 7 Comparison of calculated and measured IV curves. The V_{GS} = 9 V line is labeled for both sets of curves.